

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of : **Confirmation No. 4361**
Kenji SAITO et al. : Attorney Docket No. 2005_0635A
Serial No. 10/532,586 : Group Art Unit 3723
Filed June 17, 2005 : Examiner Robert J. Scruggs
SURFACE-TREATING PROCESS
FOR VACUUM MEMBER : **Mail Stop: Appeal Brief-Patents**

APPEAL BRIEF FILED UNDER 37 CFR § 41.37

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

THE COMMISSIONER IS AUTHORIZED
TO CHARGE ANY DEFICIENCY IN THE
FEES FOR THIS PAPER TO DEPOSIT
ACCOUNT NO. 23-0975

Sir:

The following is Appellants' Brief, submitted under the provisions of 37 CFR
§ 41.37. Pursuant to the provisions of 37 CFR § 41.20, this brief is submitted with the required
fee of \$270.00.

I. REAL PARTY IN INTEREST

The real party in interest is Nomura Plating Co., Ltd., of Osaka, Japan, the assignee of record, as recorded at Reel 016698, and Frame 0849.

II. RELATED APPEALS AND INTERFERENCES

There are no related prior or pending appeals, interferences or judicial proceedings known to Appellants, Appellants' legal representative, or assignee, which may be related to, directly affect or be directly affected by, or have a bearing on the Board's decision in the pending appeal.

III. STATUS OF CLAIMS

The status of the claims is as follows:

Claims 1, 4-11, 14 and 17-19 are pending.

Claims 1, 4-11 and 17-19 are rejected.

Claims 2, 3, 12, 13, 15 and 16 are cancelled.

Claim 14 is withdrawn.

Claims 1, 4-11 and 17-19 are appealed.

A complete copy of all of the pending claims is provided in the attached Claims Appendix.

IV. STATUS OF AMENDMENTS

The amendments set forth in the Amendment After Final Rejection, filed March 10, 2009, are entered for purposes of Appeal, as set forth in item 7 of the Advisory Action dated March 24, 2009.

V. SUMMARY OF CLAIMED SUBJECT MATTER

A concise explanation of the subject matter defined in the independent claims involved in the appeal is presented below.

Claim 1 is directed to a surface-treating process comprising:
mechanically polishing an inner surface of a vacuum member in the presence of a liquid medium, or a liquid medium and an oxidizing material,
wherein the liquid medium includes no hydrogen atom,
wherein the vacuum member is made of one kind or two or more kinds selected from the group consisting of niobium, titanium, stainless steel, copper, aluminum and iron, and
wherein the liquid medium including no hydrogen atom is a liquid at an ordinary temperature and an ordinary pressure and a saturated hydrocarbon in a molecule of which a hydrogen atom or hydrogen atoms are all substituted with a fluorine atom or fluorine atoms.

Support for this subject matter is found on page 8, lines 2-9, 16 and 17; page 8, line 28 to page 9, line 6; and page 9, lines 11-23 of Appellants' originally filed specification.

Claim 11 is directed to a forming process for a vacuum member comprising:
mechanically forming the vacuum member in the presence of a liquid medium, or a liquid medium and an oxidizing material,
wherein the liquid medium includes no hydrogen atom,
wherein the vacuum member is made of one kind or two or more kinds selected from the group consisting of niobium, titanium, stainless steel, copper, aluminum and iron, and
wherein the liquid medium including no hydrogen atom is a liquid at an ordinary temperature and an ordinary pressure and a saturated hydrocarbon in a molecule of which a hydrogen atom or hydrogen atoms are all substituted with a fluorine atom or fluorine atoms.

Support for this subject matter is found on page 8, lines 2-9, 16 and 17; page 8, line 28 to page 9, line 6; and page 9, lines 11-23 of Appellants' originally filed specification.

Claim 17 is directed to a surface-treating process comprising:

- (a) mechanically polishing an inner surface of a vacuum member in the presence of a liquid medium, or a liquid medium and an oxidizing material,
 - wherein the liquid medium includes no hydrogen atom,
 - wherein the vacuum member is made of one kind or two or more kinds selected from the group consisting of niobium, titanium, stainless steel, copper, aluminum and iron, and
 - wherein the liquid medium including no hydrogen atom is a liquid at an ordinary temperature and an ordinary pressure and a saturated hydrocarbon in a molecule of which a hydrogen atom or hydrogen atoms are all substituted with a fluorine atom or fluorine atoms; and
- (b) electrochemically polishing the inner surface of a vacuum member using an electrolytic solution including an oxidizing material.

Support for this subject matter is found on page 8, lines 2-9, 16 and 17; page 8, line 28 to page 9, line 6; page 9, lines 11-23; and page 10, lines 25-27 of Appellants' originally filed specification.

VI. GROUND OF REJECTION TO BE REVIEWED ON APPEAL

Whether claims 1, 4-11 and 17-19 are patentable under 35 U.S.C. § 103(a) over Higuchi et al., in view of Noguchi et al., Yoneda and Miller.

Whether claim 19 is patentable under 35 U.S.C. § 103(a) over Higuchi et al. in view of Noguchi et al., Yoneda, Miller and Tsuchiya et al.

VII. ARGUMENT

**Rejection of claims 1, 4-11 and 17-19 under 35 U.S.C. § 103(a) over
Higuchi et al. (JP 2000-071164) in view of Noguchi et al. (JP 11-350200),
Yoneda (JP 11-329896) and Miller (U.S. 2,951,096)**

The Position of the Examiner

The Examiner takes the position that Higuchi and Noguchi disclose the known process of surface treating an inner surface of a vacuum member by first mechanically polishing the vacuum member with a liquid medium containing hydrogen atoms, then subjecting the vacuum member to a chemical or electrochemical polishing process. The Examiner states that Higuchi and Noguchi also disclose the use of an oxidizing material formed as water which could be added to the liquid medium. However, the Examiner admits that Higuchi and Noguchi fail to teach or suggest a liquid medium absent of any hydrogen atoms where the liquid medium is a saturated hydrocarbon in a molecule of which the hydrogen atom or atoms are all substituted with a fluorine atom or atoms.

The Examiner asserts that Yoneda teach providing a solution intermingled with a polishing medium, where the non-aqueous solution is formed from various types of fluorocarbons. The Examiner states that since perfluorocarbons are examples of fluorocarbons which have had their hydrogen atoms replaced by fluorine atoms, this reference meets this limitation. Further, the Examiner states that Miller teaches that perfluorocarbons can be formed from saturated or unsaturated hydrocarbons.

The Examiner takes the position that it would have been obvious to one of ordinary skill in the art at the time the invention was made to modify the liquid medium used in the process of Higuchi and Noguchi with a liquid medium formed as a saturated hydrocarbon under ordinary pressure and ordinary temperature where they hydrogen atoms are replaced with fluorine atoms, as taught by Yoneda and Miller.

In the Advisory Action mailed March 24, 2009, the Examiner indicated that although Appellants contend that the current invention provides unexpected results when compared to the

prior art, the Examiner maintains that the combination in the rejection can be made with different motivation than described by the Appellants, and would produce the same results as disclosed by Appellants.

Appellants' Arguments

As will be demonstrated by the following arguments, the Examiner's position is untenable.

Initially, Appellants attach hereto a copy of a Comparison Table (which was previously submitted with the response filed August 19, 2008), which compares Appellants' invention with the disclosures of the cited references. Appellants assert that this table clearly demonstrates the differences between Appellants' invention and the disclosures of the cited references. The relevant disclosures of Higuchi, Noguchi and Yoneda (Japanese language) have been translated into English by Appellants, in order to prepare the attached comparison table.

As mentioned above, the Examiner admits the neither Higuchi nor Noguchi describe the liquid medium used in Appellants' claimed process, i.e., a liquid medium including no hydrogen atom, wherein the liquid medium is a saturated hydrocarbon in a molecule of which the hydrogen atom(s) are all substituted with fluorine atom(s). The Examiner relies on Yoneda as teaching the combination of a non-aqueous solution intermingled with a polishing medium, wherein the non-aqueous medium is formed from various types of fluorocarbons. However, the Yoneda reference does not teach a compound wherein **all** of the hydrogen atoms have been replaced with fluorine atoms. In fact, Yoneda teaches such compounds as hydrofluoroether and hydrofluorocarbon, which clearly include hydrogen substituents.

Specifically, Yoneda describes in lines 1-6 of paragraph [0007] that, "This invention is directed to a manufacturing method of a ceramic electronic part, comprising the step of polishing a ceramic chip . . . in a liquid. This invention is characterized by that a non-aqueous liquid is used as a liquid . . .". Further, Yoneda describes in lines 1-2 of paragraph [0014] that "barrel-polishing is typically applied in a polishing process," and in lines 6-9 of paragraph [0012] that

“[a]s a non-aqueous liquid, fluorinated inert liquids (solvents) such as hydrofluoroether, hydrofluorocarbon and chlorofluorocarbon can be most practically used.”

The Examiner appears to acknowledge that Yoneda fails to teach a compound wherein all of the hydrogen atoms have been replaced with fluorine atoms, as the Miller reference is relied upon to demonstrate that fluorocarbons exist, wherein all of the substituents are fluorine atoms. However, the Examiner has not explained why one would look from the teachings of Higuchi and Noguchi to the teachings of Yoneda or Miller.

Yoneda describes a manufacturing method of a ceramic electronic part, comprising the step of polishing a ceramic chip. (Please see paragraph [0006], lines 1-6 of Yoneda.) However, Appellants’ independent claims require polishing a vacuum member made of one kind or two or more kinds selected from the group consisting of niobium, titanium, stainless steel, copper, aluminum and iron.

The problem to be solved by Appellants’ invention is to prevent occlusion of hydrogen as a solid solution into an inner surface of the vacuum member during mechanical polishing, chemical polishing or electrochemical polishing. On the contrary, the problem to be solved by Yoneda is to prevent a ceramic electronic part from deteriorating in characteristics due to partial elution of ceramic into water at the time when a burned ceramic chip which contains alkaline earth metals such as Ba, Sr, Ca and the like is polished in water. (Please see the Patent Abstract of Japan of Yoneda, a copy of which is attached hereto. This attachment was previously submitted with the response filed August 19, 2008.) Thus, it is clear that the problem to be solved by Appellants’ invention is quite different from that of Yoneda.

Further, in the Final Office Action dated December 10, 2008, the Examiner states (on page 6, lines 11-13) that, “[t]he combination uses different motivation which is to provide a non-flammable and explosion proof solution thereby more effectively carrying out a polished process”. This statement appears to be based upon the teachings of Yoneda, i.e., paragraph [0020].

However, as mentioned previously, the object of the present invention is quite different from the above-mentioned object of Yoneda. The object of the claimed invention is to provide a novel surface-polishing process for a vacuum member capable of enhancing at a low cost a performance of

the vacuum member by suppressing occlusion of hydrogen as a solid solution into an inner surface of the vacuum member (please see page 7, lines 3-9 of the specification).

Thus, Yoneda's disclosure would not have motivated one of ordinary skill in the art to use a fluorine-based inert liquid to suppress occlusion of hydrogen as a solid solution into an inner surface of the vacuum member, as this is quite irrelevant to explosion-proof equipment.

Therefore, one of ordinary skill in the art would not have considered the combination of Yoneda with Higuchi and Noguchi.

Furthermore, Miller merely teaches that there exists a saturated or unsaturated fluorocarbon compound in which all of the substituents are fluorine atoms. However, this fact alone does not justify combining Miller with the primary references (Higuchi and Noguchi). Additionally, Miller discloses in column 2, lines 22-28 that "[a]nother object of this invention is to devise a convenient and effective synthesis ..., the perfluorocarbons and perfluorochlorocarbons especially after further treatment to saturate the unsaturated bonds present with fluorine or chlorine, being useful as lubricants where reactive substances are present." (Emphasis added.)

One of ordinary skill in the art would understand that lubricants prevent or decrease friction. On the contrary, the mechanically polishing step recited in Appellants' claims needs or makes use of friction. Thus, the Miller reference teaches away from Appellants' claimed method, and certainly cannot be relied upon as motivation to use the compound of Miller as a liquid medium in mechanical polishing. Thus, Miller fails to teach not only the element of "mechanical polishing", but also the element of "a vacuum member".

In view of the above, Appellants respectfully assert that, absent the use of Appellants' own disclosure, one skilled in the art would not have combined the references in the manner suggested by the Examiner. Appellants have studied the Miller reference quite extensively, in order to try to understand the outstanding rejections. As a result of this study, Appellants found that there is neither motivation nor a reason to combine the cited references, absent the knowledge found in Appellants' disclosure.

As stated by the Supreme Court in KSR International Co. v. Teleflex Inc., "the factfinder

should be aware, of course, of the distortion caused by hindsight bias and must be cautious of arguments reliant upon *ex post* reasoning.” (See KSR International Co. v. Teleflex Inc., 237 S. Ct. 1727 (U.S. 2007), referring to Graham v. John Deere Co. of Kansas City, 86 S. Ct. 684, which warned against a “temptation to read into the prior art the teachings of the invention in issue” and instructing courts to “guard against slipping into the use of hindsight”. For this reason, Appellants respectfully assert that the Examiner’s combination of references is unfounded, and the rejection should be withdrawn.

Furthermore, Appellants’ invention exhibits unexpected and surprising effects compared to Higuchi, which is the closest prior art. Specifically, Appellants’ invention unexpectedly suppresses the occlusion of hydrogen as a solid solution into an inner surface of the vacuum member, not only during mechanical polishing, but also during electrolytic polishing following the mechanical polishing. Due to the suppression of hydrogen occlusion, a superconducting accelerating cavity having a high performance can be made successfully, thus rendering unnecessary vacuum annealing after the polishing. Please see page 6, lines 15-22 of Appellants’ specification.

The unexpected and surprising effects of Appellants’ invention are achieved by adopting a liquid medium including a saturated hydrocarbon in a molecule of which a hydrogen atom or hydrogen atoms are all substituted with a fluorine atom or fluorine atoms when the vacuum member is formed and polished. These results would not have been expected by those of ordinary skill in the art at the time of Appellants’ invention.

Specifically, in Test Example 1 (on pages 28-30 of the present specification), the effect of various liquid media on occlusion of hydrogen as a solid solution into a plate-shaped niobium in mechanical polishing (centrifugal barrel polishing disclosed in Higuchi) was investigated. When a vacuum member is mechanically polished with water and a surfactant as a polish assistant, as disclosed in Higuchi, hydrogen is occluded as a solid solution into the vacuum member (hydrogen concentration is 79.1 ± 5.0 ppm).

On the other hand, occlusion of hydrogen as a solid solution into the vacuum member is *dramatically suppressed* by mechanically polishing the niobium sample with a liquid medium

including a saturated hydrocarbon in a molecule of which a hydrogen atom or hydrogen atoms are all substituted with a fluorine atom or fluorine atoms, such as Fluorinert FC77, as in the presently claimed invention (hydrogen concentration is 4.6 ± 0.8 ppm). Appellants direct the Examiners' attention to Test Examples 1 and 2, and Example 1 of Appellants' specification, as well as the Rule 1.132 Declaration, which was previously submitted on September 24, 2007. (A copy of this Declaration is attached hereto.) In Experiment 3 of this Declaration, the Comparative Experiment using water as a liquid medium corresponds to the invention of Higuchi, which is the closest prior art.]

Thus, Appellants' invention unexpectedly suppresses the occlusion of hydrogen as a solid solution into an inner surface of the vacuum member, not only during mechanical polishing, but also during electrolytic polishing following the mechanical polishing. Due to the suppression of hydrogen occlusion, a superconducting accelerating cavity having a high performance can be made successfully, thus rendering unnecessary vacuum annealing after the polishing. The unexpected and surprising effects of Appellants' invention are achieved by adopting a liquid medium including a saturated hydrocarbon in a molecule of which a hydrogen atom or hydrogen atoms are all substituted with a fluorine atom or fluorine atoms when the vacuum member is formed and polished. These results would not have been expected by those of ordinary skill in the art at the time of Appellants' invention.

Appellants have repeatedly presented arguments that the claimed invention results in superior and unexpected results when compared to the closest prior art. As the Examiners are certainly aware, a showing of unexpected and superior results is sufficient evidence of non-obviousness. (Please see MPEP 716.02(a).) Thus, it is asserted that Appellants' showing of superior results overcomes any asserted case of obviousness. The superior effect of Appellants' claimed invention is neither disclosed nor suggested in Higuchi, Noguchi, Yoneda, Miller, nor the combination thereof.

As mentioned above, in response to these arguments, the Examiner merely states that while Appellants provide a statement of unexpected results, the combination of references would also create this effect. In the Advisory Action, the Examiner states that the combination in the

rejection can be made with a different motivation than described by Appellants, and therefore would have the same results as disclosed by Appellants.

However, Appellants respectfully assert that the Examiner's position is untenable, as this reasoning would nullify any showing of unexpected results in any situation involving a combination of references, where the unexpected results are shown by a comparison with the prior art. Specifically, the Examiner appears to take the position that since the asserted combination of references teaches the limitations of Appellants' claimed method, then the combination of references would achieve Appellants' unexpected results. However, this position is based on an inappropriate comparison of Appellants' invention to itself, i.e., the combination of references.

As clearly explained in the MPEP, when demonstrating unexpected results, the appropriate comparison is that of Appellants' invention with the closest prior art, i.e., a single reference. MPEP 716.02(e) states, "An affidavit or declaration under 37 CFR 1.132 must compare the claimed subject matter with the closest prior art to be effective to rebut a *prima facie* case of obviousness. *In re Burckel*, 592 F.2d 1175, 201 USPQ 67 (CCPA 1979)." (Emphasis added.)

Further, section III of this portion of the MPEP states, "Although evidence of unexpected results must compare the claimed invention with the closest prior art, applicant is not required to compare the claimed invention with subject matter that does not exist in the prior art. *In re Geiger*, 815 F.2d 686, 689, 2 USPQ2d 1276, 1279 (Fed. Cir. 1987) . . . ; *In re Chapman*, 357 F.2d 418, 148 USPQ 711 (CCPA 1966) (Requiring applicant to compare claimed invention with polymer suggested by the combination of references relied upon in the rejection of the claimed invention under 35 U.S.C. 103 'would be requiring comparison of the results of the invention with the results of the invention.' 357 F.2d at 422, 148 USPQ at 714.)." Thus, the Examiner is respectfully requested to consider Appellants' showing of unexpected results, as compared to the teachings of the closest prior art, i.e., Higuchi.

Contrary to the Examiner's position, the question to consider is not whether the combination of references could or would achieve the (unexpected) results of Appellants' invention. Rather, the question is whether Appellants' invention has unexpected results when

compared to the closest prior art.

In this case, the Examiner has dismissed Appellants' showings of unexpected results. Additionally, the Examiner's rebuttal argument is based on the idea that Appellants' invention must be compared to itself. Furthermore, the Examiner has failed to assert, or provide any evidence, that the cited combinations of references would be expected to achieve the results discovered by Appellants', i.e., preventing the occlusion of hydrogen as a solid solution into a vacuum member during mechanical polishing, chemical polishing or electrochemical polishing. Thus, Appellants' showing of unexpected results demonstrates that the above-rejection should be withdrawn.

Lastly, Dr. Tamao Higuchi, who is one of the inventors of the present application, won the highest award for young researchers' presentation on the subject of the claimed invention at the 11th International Workshop on Superconducting Radio Frequency (SRF-2003), held at Travemunde, Germany, September 8-12, 2003. Please see the enclosed Japanese document regarding this award, together with its verified English translation and an English explanation of the relevant parts provided by Dr. Higuchi. The award of this prize is further evidence of the novelty and unobviousness of the claimed subject matter.

Accordingly, for the reasons set forth above, neither the subject matter of Appellants' claims, nor the unexpected and superior effects resulting therefrom, is taught or suggested by Higuchi, Noguchi, Yoneda, or Miller, or the cited combination thereof. Thus, it is respectfully requested that the above-rejection be withdrawn.

**Rejection of claim 19 under 35 U.S.C. § 103(a) over
Higuchi et al. (JP 2000-071164) in view of Noguchi et al. (JP 11-350200),
Yoneda (JP 11-329896), Miller (U.S. 2,951,096) and Tsuchiya et al. (U.S. 2002/0093002)**

The Position of the Examiner

The Examiner's position regarding Higuchi in view of Noguchi, Yoneda and Miller is previously discussed. The Examiner admits that these references lack an oxidizing agent formed as nitric acid. The Examiner relies on Tsuchiya only to assert that it is old and known in the art to provide an oxidizing agent with nitric acid.

Appellants' Arguments

Claim 17 is patentable over Higuchi, Noguchi, Yoneda, and Miller for the reasons set forth in detail above. Claim 19 depends directly upon claim 17, and is thus patentable over these references for the same reasons claim 17 is patentable over these references.

As mentioned above, Tsuchiya is only relied upon to assert that it is old and known in the art to provide an oxidizing agent with nitric acid. However, Tsuchiya fails to remedy the deficiencies of the remaining references. Thus, claim 19 is patentable over the cited combination of references for the reasons previously stated.

Accordingly, for the reasons set forth above, neither the subject matter of Appellants' claims, nor the unexpected and superior effects resulting therefrom, is taught or suggested by Higuchi, Noguchi, Yoneda, Miller, or Tsuchiya, or the cited combination thereof. Thus, it is respectfully requested that the above-rejection be withdrawn.

Conclusion

For the foregoing reasons, the invention of claims 1, 4-11 and 17-19 is patentable over the combinations of references relied upon by the Examiner. Thus, reversal of the final rejection is respectfully requested.

Attached hereto are a Claims Appendix, an Evidence Appendix and a Related Proceedings Appendix.

The brief is submitted with the required fee.

Respectfully submitted,

Kenji SAITO et al.

/Amy E. Schmid/

By: 2009.07.10 13:20:15 -04'00'

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VIII. CLAIMS APPENDIX

1. (Appealed) A surface-treating process comprising:

mechanically polishing an inner surface of a vacuum member in the presence of a liquid medium, or a liquid medium and an oxidizing material,

wherein the liquid medium includes no hydrogen atom, wherein the vacuum member is made of one kind or two or more kinds selected from the group consisting of niobium, titanium, stainless steel, copper, aluminum and iron, and

wherein the liquid medium including no hydrogen atom is a liquid at an ordinary temperature and an ordinary pressure and a saturated hydrocarbon in a molecule of which a hydrogen atom or hydrogen atoms are all substituted with a fluorine atom or fluorine atoms.

2-3. (Cancelled)

4. (Appealed) The surface-treating process according to claim 1, wherein the vacuum member is made of niobium.

5. (Appealed) The surface-treating process according to claim 1, wherein the vacuum member is a superconducting accelerating cavity.

6. (Appealed) The surface-treating process according to claim 1, wherein the mechanical polishing is performed in the presence of an oxidizing material.

7. (Appealed) The surface-treating process according to claim 6, wherein the oxidizing material is ozone, a mixture of ozone and oxygen, or hydrogen peroxide water.

8. (Appealed) The surface-treating process according to claim 1, wherein after the mechanical polishing, the inner surface of a vacuum member is subjected to chemical polishing or electrochemical polishing.

9. (Appealed) The surface-treating process according to claim 1, wherein after the mechanical polishing, the inner surface of a vacuum member is subjected to electrochemical polishing using an electrolytic solution including an oxidizing material.

10. (Appealed) The surface-treating process according to claim 9, wherein the oxidizing material is ozone, hydrogen peroxide water or nitric acid.

11. (Appealed) A forming process for a vacuum member comprising:

mechanically forming the vacuum member in the presence of a liquid medium, or a liquid medium and an oxidizing material,

wherein the liquid medium includes no hydrogen atom, wherein the vacuum member is made of one kind or two or more kinds selected from the group consisting of niobium, titanium, stainless steel, copper, aluminum and iron, and

wherein the liquid medium including no hydrogen atom is a liquid at an ordinary temperature and an ordinary pressure and a saturated hydrocarbon in a molecule of which a hydrogen atom or hydrogen atoms are all substituted with a fluorine atom or fluorine atoms.

12-13. (Cancelled)

14. (Withdrawn) An electrolytic polishing solution including an oxidizing material and used in electrochemical polishing of a vacuum member.

15-16. (Cancelled)

17. (Appealed) A surface-treating process comprising:

(a) mechanically polishing an inner surface of a vacuum member in the presence of a liquid medium, or a liquid medium and an oxidizing material,

wherein the liquid medium includes no hydrogen atom,

wherein the vacuum member is made of one kind or two or more kinds selected from the group consisting of niobium, titanium, stainless steel, copper, aluminum and iron, and

wherein the liquid medium including no hydrogen atom is a liquid at an ordinary temperature and an ordinary pressure and a saturated hydrocarbon in a molecule of which a hydrogen atom or hydrogen atoms are all substituted with a fluorine atom or fluorine atoms; and

(b) electrochemically polishing the inner surface of a vacuum member using an electrolytic solution including an oxidizing material.

18. (Appealed) The surface-treating process according to claim 17, wherein the oxidizing material is ozone, hydrogen peroxide water or nitric acid.

19. (Appealed) The surface-treating process according to claim 17, wherein the oxidizing material is nitric acid.

IX. EVIDENCE APPENDIX

1. Comparison Table of the present invention with the cited references, previously submitted with response filed August 19, 2008.
2. Abstract of JP 11-329896, previously submitted with response filed August 19, 2008.
3. Declaration Under 37 CFR 1.132, of Kenji Saito, previously submitted with response filed September 24, 2007.
4. Article entitled “Dr. Tamao HIGUCHI Won the Highest Award for Young Researcher’s Presentation”, <http://www.kck.jp/intra-j/news/2003/srf-2003-j.html>, together with English translation, and explanation of relevant parts by Dr. Higuchi.

X. RELATED PROCEEDINGS APPENDIX

None

Comparison Table of the present invention with the cited references

The present invention Priority date: Dec. 6, 2002	D1: Higuchi (JP 2000-71164) Pub. date: Mar. 7, 2000	D2: Noguchi (JP 11-350200) Pub. date: Dec. 21, 1999	D3: Yoneda (JP 11-329896) Pub. date: Nov. 30, 1999	D4: Miller (US 2,951,096) Pub. date: Aug. 30, 1960
A surface-treating process,	a method for polishing physically the metal hollow object like the acceleration cavity having openings at the both ends (para.[0006], lines 2-3)	To provide a method for polishing the inside surface of a metallic hollow body (Abstract, line 2-3)	the ceramic chip surface such as end face is shaved off by this polishing (para.[0011], lines 6-8)	No mention
wherein an inner surface of a vacuum member is mechanically polished	a method for polishing physically the metal hollow object like the acceleration cavity having openings at the both ends (para.[0006], lines 2-3)	No mention	In a polishing process, barrel-polishing is typically applied (para.[0014], lines 1-2)	No mention
		The process for chemical polishing or electrolytic polishing the inside surface of the metal hollow object --- (para.[0006], lines 4-5)	This invention is directed to a manufacturing method of a ceramic electronic part, comprising the step of polishing a ceramic chip after calcination in a liquid (para.[0007], lines 1-4)	
in the presence of a liquid medium, or a liquid medium and an oxidizing material,	the existence of a polish assistant (water, surfactant) etc. influenced the amount of polish removal (para.[0006], lines 8-9)	the polish liquid is characterized by --- at least one of fluoric acid, sulfuric acid, nitric acid, phosphoric acid, chromic anhydride, sodium hydroxide, and sodium phosphate (para.[0008], lines 4-7)	This invention is directed to a manufacturing method of a ceramic electronic part, comprising the step of polishing a ceramic chip --- in a liquid (para.[0007], lines 1-4)	No mention
wherein the liquid medium includes no hydrogen atom, and	No mention	No mention	No mention	Another object of this invention is to produce substantially hydrogen-free fluorocarbons --- (col.1, lines 66-67)
wherein the liquid medium including no hydrogen atom is a liquid at an ordinary temperature and an ordinary pressure and	No mention	No mention	No mention	--- useful compounds are made by heating at a temperature in the range of about 90°C. to about 700°C., a compound --- (col.3, lines 47-50); The reaction is suitably carried out at atmospheric pressure (col.4, lines 1-2)
a saturated hydrocarbon in a molecule of which a hydrogen atom or hydrogen atoms are all substituted with a fluorine atom or fluorine atoms.	No mention	No mention	No mention	The term "fluorocarbon" designates a carbon compound in which all of the substituents are fluorine ---. Such a compound may be saturated or unsaturated (col.1, lines 26-32); a perhalogenated compound is a compound in which all the hydrogen atoms are replaced by halogen (col.1, lines 38-40)
		In this invention, fluorinated inert liquids can be preferably used as a non-aqueous liquid ([0008], lines 3-4); As a non-aqueous liquid, fluorinated inert liquids (solvent) such as hydrofluoroether, hydrofluorocarbon and chlorofluorocarbon can be most practically used (para.[0012], lines 6-9)		

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 11-329896

(43)Date of publication of application : 30.11.1999

(51)Int.Cl.

H01G 4/30

B24B 31/00

C04B 41/91

H01G 4/12

(21)Application number : 10-134760

(71)Applicant : MURATA MFG CO LTD

(22)Date of filing : 18.05.1998

(72)Inventor : YONEDA YASUNOBU

(54) MANUFACTURE OF CERAMIC ELECTRONIC PART

(57)Abstract:

PROBLEM TO BE SOLVED: To prevent a ceramic electronic part from deteriorating in characteristics due to partial elution of ceramic into water at the time when a burned ceramic chip which contains alkaline earth metals such as Ba, Sr, Ca and the like is polished in water.

SOLUTION: A burned ceramic chip is polished in a non-aqueous liquid so as to prevent the ceramic chip from being adversely influenced by water. Fluorine-based inert liquid such as hydrofluoroether, hydrofluorocarbon, or chlorofluorocarbon is especially favorably used as non-aqueous liquid.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of : Confirmation No. 4361
Kenji SAITO et al. : Group Art Unit: 3723
Serial No. 10/532,586 : Examiner: Robert J. Scruggs
Filed: April 25, 2005

DECLARATION UNDER 37 CFR 1.132

Honorable Commissioner of Patent and Trademarks

Sir:

I, Kenji SAITO declare that:

I was born in Ehime Prefecture, Japan, on April 6, 1952;

I am an inventor of the above-identified US patent
Application;

I am a citizen of Japan and a resident of 27-14, Umezono
2-chome, Tsukuba-shi, Ibaraki-ken 305-0045, Japan;

I graduated from Ritsumeikan University, Science and
Technology Department, Kyoto-shi, Kyoto-fu, Japan in 1978;

I took the doctor degree on the study of the nuclear
physics at Tohoku University, Sendai-shi, Miyagi-ken, Japan in
March 1983. My doctoral work dealt the nuclear magnetic
structure of ^{24}Mg entitled "Inelastic magnetic transition from
 ^{24}Mg used 180° electron scattering";

I was appointed as researcher at Japan Society for the
Promotion of Science (Nippon Gakujutsu Shinkou-kai) in 1983 and
worked for two years 1983-1985 at High Energy Accelerator

Research Organization, 1-1 Oho, Tsukuba-shi, Ibaraki-ken, Japan. I innovated Horizontal Electropolishing Method for superconducting RF niobium cavity there;

I was appointed as associated researcher at High Energy Accelerator Research Organization in 1985. I contributed to the construction of a large superconducting RF accelerator system in the TRISTAN project, which was realized firstly in the world;

I was promoted to assistant professor position at High Energy Accelerator Research Organization in November 2000;

I made concurrently serving the Nagoya University in for one year in 2004. I made lecture there on Superconducting RF cavities for graduate students;

I am concurrently serving the Tokyo University. I am lecturing on Superconducting RF cavities for graduate students since September 2006;

I visited Thomas Jefferson Accelerator Laboratory (JLAB), Newport News, Virginia, U.S.A. during April 1990 to March 1992 as a first guest scientist of JLAB. I was invited by Professor R. Sundelin and worked with Professor. P. Kneisel. I contributed the start-up of CEBAF accelerator construction. I educated CEBAF's engineers and technicians how to assemble cavities in clean-room and to measure them at cryogenic temperature. I developed high gradient SRF cavities with a 30MV/m gradient;

I visited JLAB again in 1994 by short term visiting fellowship of Japanese Ministry of Education and Technology for a half of year in order to study cryogenic refrigerator system;

I studied on the nuclear physics including below 1) to 4);

I switched to accelerator science and technology since

1983 as mentioned above;

The papers below 5) to 118) are on R&D of accelerator.

I reported the following papers up to 2004;

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- 8) Takaaki Furuya, Kiyomitsu Asano, Yuzo Kojima, Shinji Mitsunobu, Hirotaka Nakai, Toshiharu Nakazato, Shuichi Noguchi, Kenji Saito and Tsuyoshi Tajima: " The TRISTAN Superconducting Cavities ", Proc. of the 3rd Workshop on RF Superconductivity, ANL, U.S.A., September, 1987, pp.95 - 108.
- 9) S.Noguchi, K.Akai, M.Arinaga, K.Asano, T.Furuya, K.Hara, K.Hosoyama, A.Kabe, Yuji Kojima, Yuzo, Kojima, S.Mitsunobu, H.Nakai, T.Nakazato, T.Ogitsu, K.Saito, U.Sakamoto, T.Suzuki and T.Tajima: "Status of TRISTAN Superconducting RF Program ", Proc. of the 3rd Workshop on RF Superconductivity, ANL, U.S.A., September, 1987, pp. 605 - 624.

1988 -

- 10) Kiyomitsu Asano, Takaaki Furuya, Yuzo Kojima, Shinji Mitsunobu, Hirotaka Nakai, Shuichi Noguchi, Kenji Saito and Tsuyaoshi Tajima: " XPS and AES Studies of Thin Oxide Layers on Niobium for Superconducting RF Cavities ", KEK Report 88 - 2, 1988.

1989 -

- 11) Y.Kojima, K.Akai, M.Arinaga, K.Asano, E.Erura, T.Furuya, K.Hara, K.Hosoyama, A.Kabe, E.Kako, Y.Kojima, K.Kobo, S.Kurokawa, S.Mitsunobu, H.Nakai, T.Nakazato, S.Noguchi, T.Ogitsu, K.Saito, Y.Sakamoto, T.Shishido, TSuzuki, T.Tajima and T.Takahashi: " Upgrading of TRISTAN by Superconducting RF System ", Proc. of 1989 Particle Accelerator Conference, Chicago, Illinois, U.S.A., 1989, pp. 1789 - 1791.

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The experiments set out below were conducted under my supervision and direction.

Experiment 1

Comparative investigation on occlusion of hydrogen as a solid solution according to a kind of a liquid medium in centrifugal barrel polishing

(1) Test method

Dehydrogenation of an L band niobium single cell cavity (a length of 370 mm and the maximum diameter of 210 mm) was conducted by applying vacuum annealing at 750°C for 3 hours thereto. Inserted into the cavity was a plate-shaped niobium sample (a thickness of 2.5 mm, a width of 1 mm and a length in the range from 147 to 149 mm, which is also simply referred to as a sample) dehydrogenated in a similar way and thereafter an inner surface of the cavity and the niobium sample were subjected

to centrifugal barrel polishing using Fluorinert™ fluorine containing inert liquid (Fluorinert™) FC-77 (a mixture of $C_8F_{16}O$ and C_8F_{18}) manufactured by 3M Co. as a liquid medium with a resulted average polishing-off thickness of about 30 μm . Note that a polishing-off thickness of 30 μm corresponds to a thickness of an affected layer on a surface of niobium material to be removed by the polishing judging based on experiments in the past and the rule of thumb. Centrifugal barrel polishing was performed in conditions described in Table 1 with the apparatus shown in Figs. 1 and 2.

Table 1

Rotation number	160 rpm
Revolution number	160 rpm
Polishing chips	GCT
Amount of polishing chips	2000 cm^3
Amount of liquid medium	850 ml
Polishing time	4 hrs

Fig. 1

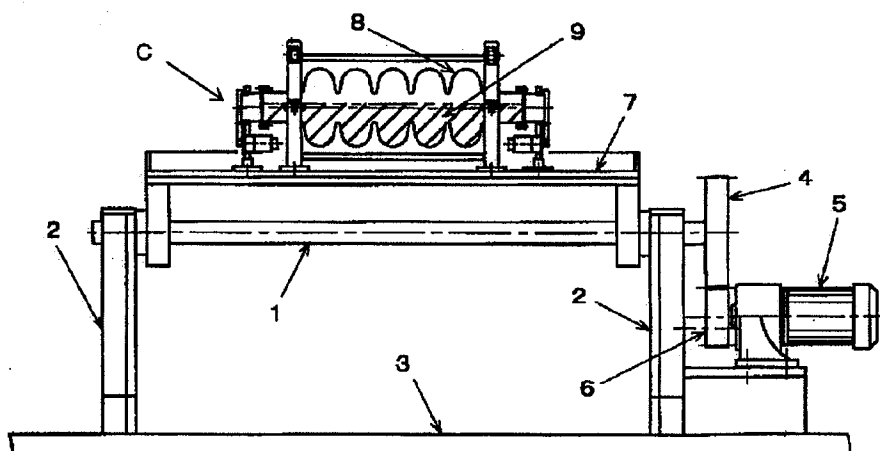
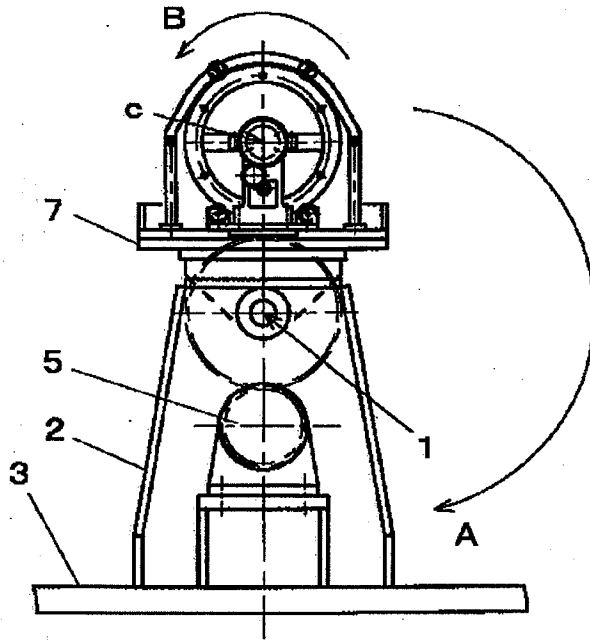


Fig. 2



Note that triangular prism-shaped GCT containing silicon carbide (SiC) as abrasive grains (manufactured by TKX Co.) was adopted as polishing chips. Furthermore, for comparison, prepared from the same material were a sample obtained by centrifugal barrel polishing in a dry condition without using a liquid medium, a sample obtained by centrifugal barrel polishing using a mixture of water and a surfactant as a liquid medium and a sample obtained by centrifugal barrel polishing using a hydrogen peroxide water or absolute propyl alcohol as a liquid medium.

Measurements were conducted on polishing-off thickness of the polished niobium samples and hydrogen concentrations in the samples. A polishing-off thickness was measured with an ultrasonic film thickness meter (manufactured by NOVA Co. with a model 800+). A hydrogen concentration in a sample was measured

with RH-1E method of LECO Co. (a combination of an inert gas melting method and a thermal conductivity method described in JIS-Z-2614).

(2) Test result

Results of the measurements are shown in Table 2.

Table 2

Liquid medium	Hydrogen concentration (detected values: ppm)	Polishing-off thickness (μm)
Water + Surfactant	79.1 ± 5.0	About 30
None (dry)	10.9 ± 0.8	About 0 to 5
Absolute propyl alcohol	49.4 ± 2.2	About 30
Hydrogen peroxide water (10%)	28.4 ± 1.4	About 30
Fluorinert FC-77	4.6 ± 0.8	About 30

An average polishing-off thickness in the range from about 0 to 5 μm in a case of a dry polishing (without a liquid medium) shows almost no polishing-off on the sample in the method. From the results, it was made clear that mechanically polishing with Fluorinert FC-77 having no hydrogen atom in a molecule thereof as a liquid medium greatly suppresses occlusion of hydrogen as a solid solution into a member to be polished in comparison with other liquid media.

Experiment 2

Comparative investigation on occlusion of hydrogen as a solid solution with ozone contained in liquid medium in centrifugal

barrel polishing

(1) Test method

After a plate-shaped niobium sample (a thickness of 2.5 mm, a width of 1 mm and a length in the range from 147 to 149 mm), according to Experiment 1, was put into an L band niobium single cell cavity (a length of 370 mm and the maximum diameter of 210 mm) dehydrogenated by vacuum annealing, the sample was subjected to centrifugal barrel polishing with FC-77 alone or a mixture of FC-77 and ozone in which ozone is absorbed in FC-77, as a liquid medium.

(2) Test result

Hydrogen concentrations (ppm) in samples after the central barrel polishing are as shown in Table 3.

Table 3

Liquid medium	Hydrogen concentration (ppm)	Polishing-off thickness (μm)
FC-77 alone	4.60 ± 0.8	about 30
FC-77 + ozone	2.67 ± 0.5	about 30

From Table 3, it was found that hydrogen concentrations in samples are both low, if they are subjected to centrifugal barrel polishing with FC-77 alone and mixture of FC-77 and ozone, respectively, as a liquid medium, which means that occlusion of hydrogen as a solid solution into the samples was remarkably suppressed during centrifugal barrel polishing.

Experiment 3

Manufacture of niobium superconducting accelerating cavity

(1) Test method

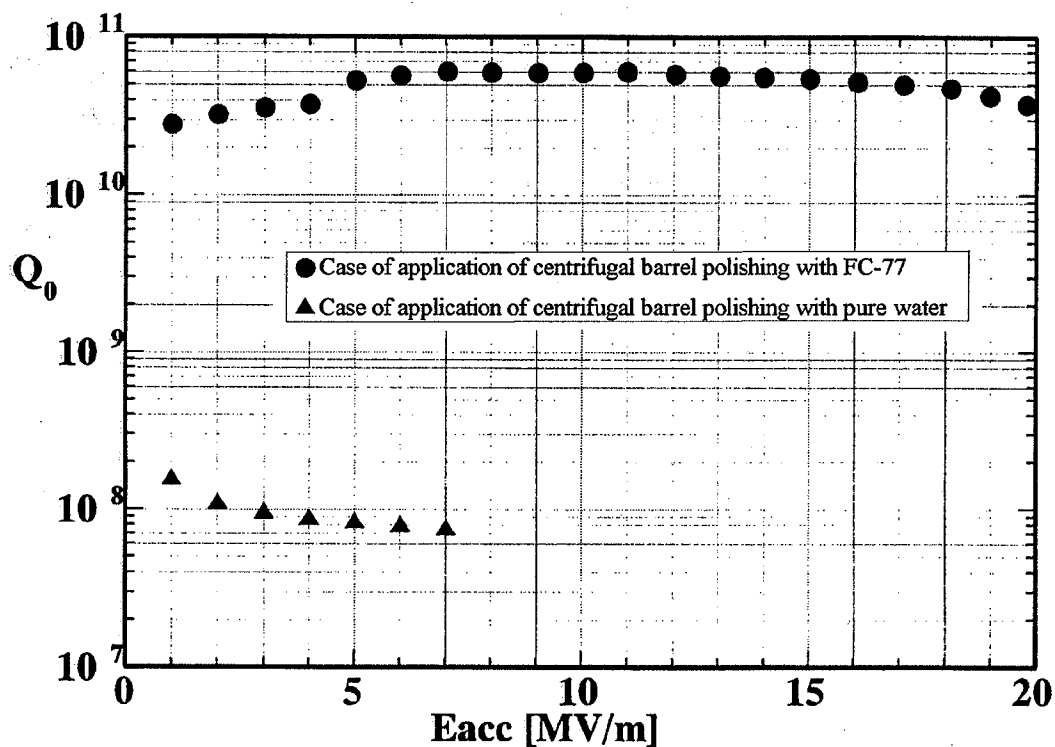
Installed in the apparatus of Fig. 1 was a 1300 MHz single cell cavity with a total cavity length of 370 mm, the maximum cavity diameter of 210 mm, a beam pipe diameter of 80 mm and a thickness of 2.5 mm and the single cell cavity was subjected to centrifugal barrel polishing. Conditions for centrifugal barrel polishing were in conformity with those of Experiment 1 and FluorinertTM fluorine containing inert liquid (FluorinertTM) FC-77 manufactured by 3M Co. was employed as a liquid medium. After cleaning with pure water, the cavity was placed on a support table with a rotation activating function and an inverting function, a chemical polishing solution kept at 30°C and composed of 89 w/v % phosphoric acid : 67 w/v % nitric acid : 40 w/v % hydrofluoric acid = 1 vol : 1 vol : 1 vol was continuously fed at a flow rate of 10 L/min through the cavity while the cavity was rotated at 10 rpm, and chemical polishing was thus conducted in the cavity for 10 minutes (a target of polishing-off was 50 μ m) as shown in Fig. 2. Thereafter, while the cavity is rotated, the polishing solution was rapidly discharged and, also, rolling and inverting were alternately effected in a repeated manner to clean the cavity by means of a common method. As Comparative Experiment, another single cell cavity was subjected to centrifugal barrel polishing with water only as a liquid medium and then a single cell cavity chemically polished in conformity with the above-mentioned

procedure.

(2) Test result

Total polishing-off thickness values of the cavities of Experiment 3 and Comparative Experiment thus obtained were measured with the result of an average thickness of about 80 μm . Acceleration performances (Q-values and accelerating electric fields [E_{acc} : MV/m]) of the cavities are shown in Fig. 3.

Fig. 3



Note that a measurement test for an acceleration performance was conducted at 1.4 K to which the cavity was cooled after being held at 100K for 16 hours in order to clearly confirm reduction

in Q-value due to occlusion of hydrogen as a solid solution. Reduction in Q-value was observed with a rise in an accelerating electric field in the cavity of Comparative Experiment obtained in a procedure in which after centrifugal barrel polishing with pure water, chemical polishing was applied, whereas no reduction in Q-value in the cavity of Experiment 3 was observed even with a rise in accelerating electric field. Therefore, it is clear that the accelerating cavity manufactured in Experiment 3 has by far higher acceleration performance as compared with that manufactured in Comparative Experiment.

It is declared by the undersigned that all statements made herein of undersigned's own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001, and that such willful false statements may jeopardize the validity of the above-identified application or any patent issuing thereon.

This 14th day of September, 2007

Kenji Saito

Kenji SAITO



樋口玉緒氏 若手研究発表最優秀賞受賞

9月8日(月)から9月12日(金)にかけて、ドイツ・トラベミュンデで開催された第11回超伝導応用国際会議(Workshop on RF Superconductivity, SRF-2003)において、樋口玉緒氏が、若手研究発表(20分のオーラル)最優秀賞(初代)を受賞しました。

樋口氏は、今年3月「新しい機械研磨と電解研磨による水素吸蔵を起こさない超伝導空洞の表面処理法の開発」で総合研究大学院大学(総研大)博士号(工学博士)を取得しました。その内容を発表したもので、研究のオリジナリティー、超伝導空洞業界への多大な寄与(簡単、低コストの表面処理)が高く評価されました。

同氏の研究略歴

- 1994年3月 立命館大学大学院・理工学研究科・物理学専攻・博士課程前期課程終了。理学修士。
- 1994年4月 株式会社 野村鍍金入社。
- 1994年5月より2年間KEK受託研究員として超伝導空洞の表面処理の研究に従事。
特に、ニオブ空洞の内面機械研磨として簡便なパレル研磨の開発を行う。



- 1997年4月 株式会社野村鍍金鹿沼工場でニオブ加速空洞の電解研磨及び化学研磨等の表面処理技術業務につく。
- 1999年4月、高速パレル研磨の開発のためKEKの要請を請け、再度KEK受託研究員。一貫した研究活動のために、同年、9月総研大を受験、合格。
- 2000年4月～2003年3月会社在籍のまま、社会人学生として総研大在学。遠心パレル研磨の開発、電解研磨での水素吸蔵に関する研究に専念。
- 2003年3月「新しい機械研磨と電解研磨による水素吸蔵を起こさない超伝導空洞の表面処理法の開発」の研究で総研大・博士号(工学博士)を取得。
- 2003年9月 SRF2003での若手研究者発表最優秀賞(初代)を受賞。

SRF2003での発表内容

総研大での博士研究結果として、SRF2003で「水素フリー電解研磨と水素吸蔵現象について」の題目で超伝導ニオブ加速空洞の表面処理に関する発表を行った。発表の内容は以下の通りである。

超伝導加速空洞の大規模実用化は1989年にKEKに建設されたTRISTAN超伝導空洞で初めて成功した。この成功以後、超伝導加速空洞が各国の研究所で電子・陽電子衝突型のストレージリング、FELなど広く用いられるようになった。次世代の超伝導加速空洞の大規模応用として、例えばTESLA(TeV Energy Superconducting Large Accelerator)がある。TESLAでは25MV/m以上(従来の5倍)の高電界性能が要求される。この大規模応用では空洞の加速電界の向上と同時に空洞製作コスト削減が重要な研究課題である。特に、空洞製作面では表面処理における空洞性能の高信頼性の確保と同時に処理工程の単純化が必須である。

TRISTANは、長期運転における超伝導空洞の当時の高性能(5-10MV/m、 $Q_0=2 \times 10^9$)、高信頼性を実証した。しかし、その表面処理工程は、機械研磨(パフ研磨)、予備電解研磨(2-3 μm)、電解研磨(80 μm)、真空アニール、仕上げ電解研磨(10 μm)と煩雑・高コ

ストであった。ここでバフ研磨は空洞内面の表面欠陥層を強力に除去する工程であり、空洞性能の信頼性を上げるために重要な工程である。しかし、このバフ研磨では、砥粒をニオブ面に埋め込む。電解研磨液循環システムを砥粒で汚染しないように、予めその汚染を除去する必要がある。そのために、予備電解研磨が必要で、空洞内に閉じ込めた研磨液で少量電解研磨し、その汚染液を捨てる方法をとっている。電解研磨は、滑らかな表面を作るもので、当時としてはKEKのみが採用した方法である。最近では、高電界空洞にとって不可欠の技術として世界的に注目されている。真空アニールは電解研磨処理中に吸蔵された水素を除去し、水素吸蔵による空洞の性能劣化(水素病)を回復させる工程である。しかし、工業炉を使用するので空洞内表面が汚染され、表面を清浄にするために仕上げ電解研磨が必要であった。

既に述べたように、TRISTANでは機械研磨としてバフ研磨を採用した。空洞は、ハーフセルを電子ビーム溶接して製作するが、その溶接シーム部には、溶接欠陥が発生し易く、それがクエンチの原因になるので、その部分をも積極的に研磨する必要がある。しかし、バフ研磨は多セル化された複雑空洞形状に適応することが難しく電子ビーム溶接シームを一括研磨できない。つまり、空洞完成後、溶接シームは別途グランド研磨しなければならない。そこで、一括研磨可能な簡単な機械研磨方法としてバレル研磨を開発した。この方法は、空洞内に研磨石と研磨剤(石鹼水)を空洞内に入れてガラガラ回して研磨する方法であり、時間がかかるが非常に簡便である。この方法で空洞性能が確保出来ることを確認した上で、バレル研磨の低研磨速度の克服のために、遠心バレル研磨法(公知の技術)を適用し、研磨速度の高速化に成功した。9連空洞を例にとると、バフ研磨と溶接部のグランド研磨で3週間程度を要していたところを1日に短縮できた。

この機械研磨の後、研磨砥粒の埋め込みや研磨カスを除去するため化学研磨或いは電解研磨(予備研磨)を施す必要がある。しかし、高電界性を確保するには化学研磨ではなく電解研磨による滑らかな表面仕上げが不可欠である。我々は機械研磨と電解研磨の組合せ処理を基本とした簡単な表面処理の開発に着手した。つまり、アニールを省くことを追求した。

始めに遠心バレル研磨と電解研磨を施し真空アニールを省いたところ、結果は著しい水素病が発生した。

次に、機械研磨を施さずにバンド単セル空洞を横型連続電解研磨(TRISTANの方法)で200 μ m多量研磨し、水素病が観察されなかった。従来から電解研磨で水素吸蔵し易いことが知られているが、ここでは電解研磨は必ずしも水素吸蔵を引き起こすとは限らないことを突止め、更に電解研磨中、常時電圧を印加することが水素吸蔵防止に有効であることを明確にした。つまり、電解研磨では、空洞が陽極になるので、サイズの小さい水素イオン(H^+)がポテンシャルバリアーのためにニオブに侵入できない。そこで機械研磨工程が水素吸蔵の原因と考え、サンプルを用いて水素ガス分析調査した。遠心バレル研磨で多量の水素吸蔵が起きること、バレル研磨液(水等)を構成する水素がニオブに吸蔵されることが分かった。水素を含まない溶媒(市販のフロリナート)の選択でこの水素吸蔵の問題を解決し、水素フリーの遠心バレル研磨法の開発に成功した。これが、一つ目の発明である。

この水素フリー遠心バレル研磨と電解研磨、或いは化学研磨を組合せた表面処理法をバンド単セル空洞に適用した。電解研磨を組合せた場合には著しい水素病が発現したが、化学研磨を組合せた場合には水素病が起らなかった。

電解研磨を組合せた場合の失敗から、機械研磨キズのあるニオブ表面が特に水素を吸蔵しやすいことが分かった。また、化学研磨を組合せた場合の成功から、酸化プロセスが水素吸蔵阻止に重要であることが示唆された。化学研磨では定常的な酸化過程とその酸化物の溶解により研磨が進行する。電解研磨では印加電圧による電気化学的な酸化過程とその酸化物の溶解により研磨が進行する。予備電解研磨では、機械研磨による表面欠陥が多数存在する状態で印加電圧がかけられない状況を経由する(研磨液注入時等)。このプロセスに於いて水素陽イオンが表面欠陥に吸蔵されることをサンプルで確認した。また、表面の機械研磨キズを除去した後では、その後の電解研磨で水素を吸蔵しない。したがって、電解研磨でも定常酸化過程を付加すれば予備電解研磨

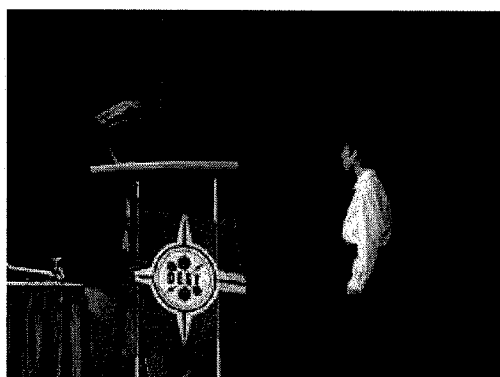
で起る水素吸蔵を防止できる可能性がある。予備電解研磨液に酸化剤である硝酸添加を試みた。ここで硝酸添加量が多いと化学研磨になり電解研磨面が得られない。硝酸添加量が少量であることが望ましい。1500ppm(研磨液4リットルに対し60%濃度の硝酸10cc)の少量硝酸添加をした電解研磨液でLバンド単セル空洞を予備研磨し、その後従来法による連続電解研磨を50 μ m施した。水素病は起こらず、加速電界30MV/mが達成できた。その再現性を確認できた。これが二つ目の発明である。

結論として、

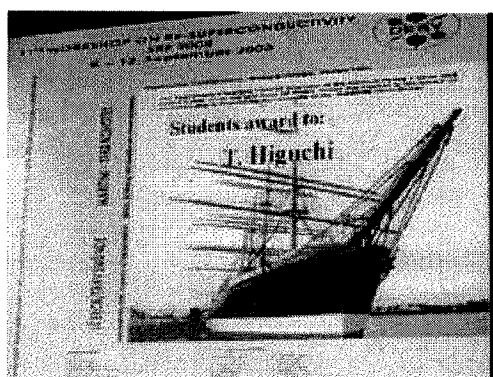
1. ニオブ空洞の簡便かつ高速な機械研磨法(遠心バレル研磨法)を開発した。
2. この機械研磨法での水素吸蔵を調査し、水素フリー機械研磨法を発明した。
3. 定常酸化プロセスを付加した電解研磨法(もちろん化学研磨でも可)で遠心バレルによる砥粒汚染層、ダメージ層を水素吸蔵させることなく除去する方法を発明した。
4. こうした発明の組み合わせと従来の電解研磨を採用することで30MV/mの高電界性を保証する簡単な表面処理法を確立した。
5. 結果としてアニール不要で簡単な超伝導空洞表面処理法を開発した。

この開発の中で、湿式機械研磨では金属表面に研磨キズが発生する瞬間にそのキズが研磨液を構成する水素を分解・吸着・吸蔵するラジカル反応が起きることを明確にした。また、定常酸化プロセスがこのラジカルな水素吸蔵プロセスの進行を阻止するのに有効であることを見出した。

この研究成果は、超伝導空洞の表面処理を著しく簡単化するもので、TESLAのように超伝導空洞を2万台以上も製造する場合、設備投資の低減、処理時間の短縮化等大きなコストメリットを導くことは明らかである。



(拡大写真 36KB)



(拡大写真 36KB)

- ◆ [研究者向けトップページ](#)
- ◆ [共同利用研究者>G正職員専用ページ](#)

VERIFICATION OF TRANSLATION

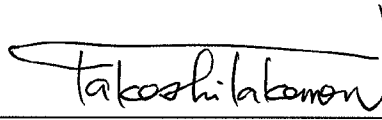
I, Takeshi Takemori, of 1-2-512, Denpo 1-chome, Osaka-shi, OSAKA 554-0002 JAPAN, state the following:

I am fluent in both the English and Japanese languages and capable of translating documents from one into the other of these languages.

The attached document is a true and accurate English translation to the best of my knowledge and belief of the relevant portions of "Dr. Tamao HIGUCHI Won the Highest Award for Young Researches' Presentation" (<http://www.kek.jp/intra-j/news/2003/srf-2003-j.html>).

I state that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true.

Signature:



Takeshi Takemori

Date:

June 4, 2009

Dr. Tamao HIGUCHI Won the Highest Award for Young Researches' Presentation

(Page 1, the 1st-2nd paragraphs)

Dr. Tamao Higuchi won the highest award for young researchers' presentation (oral, 20 minutes) (the first winner) at the 11th International Workshop on Superconducting Radio Frequency (Workshop on RF Superconductivity, SRF-2003) held at Travemuende, Germany, September 8-12, 2003.

Dr. Higuchi took the doctor degree (engineering) at the Graduate University for Advanced Studies, Japan, in March this year. Her doctoral work dealt with "Development of surface treating process for superconducting cavity with no hydrogen occlusion by novel mechanical polishing and electropolishing". Dr. Higuchi presented her doctoral work. The originality of Dr. Higuchi's study and its great contribution to the field of superconducting cavity (simple surface-treating process at low cost) were highly evaluated.

(Page 1, the 4th paragraph)

The contents of Dr. Higuchi's presentation at SRF 2003

Dr. Higuchi presented her doctoral study on the surface treating of superconducting niobium cavity entitled "DEVELOPMENT OF HYDROGEN-FREE ELECTROPOLISHING AND HYDROGEN OCCLUSION PHENOMENA". The contents of Dr. Higuchi's presentation are as follows.

(Page 2, lines 30-43)

Firstly, centrifugal barrel polishing and electropolishing were carried out without vacuum annealing. As a result, heavy hydrogen disease occurred.

Subsequently, an L-band single-cell cavity was subjected to horizontal continuous electropolishing (TRISTAN's process) removing a large thickness of 200 μm . As a result, hydrogen disease was not observed. Conventionally, it is known that

electropolishing easily causes occlusion of hydrogen. In the present study, it has been confirmed that electropolishing does not necessarily cause occlusion of hydrogen. Further, it has been made clear that it is effective for preventing occlusion of hydrogen to constantly apply electric voltage. That is, the cavity becomes anode in electropolishing and therefore a hydrogen ion (H^+) which is small in size cannot penetrate niobium sample because of potential barrier.

Based on the above-mentioned results, it was considered that mechanical polishing step causes occlusion of hydrogen. After analyzing and investigating hydrogen gas in samples, it was determined that centrifugal barrel polishing causes a large amount of hydrogen occlusion and a hydrogen atom in barrel polishing liquid (water etc.) is occluded by a niobium sample. This problem of hydrogen occlusion was solved by selecting a solvent which does not include a hydrogen atom (Fluorinert, commercially available). The development of hydrogen-free centrifugal barrel polishing process was succeeded. This is the first invention.

(Page 3, lines 8-21)

In conclusion,

1. Simple and fast mechanical polishing process (centrifugal barrel polishing) for niobium cavity was developed.

2. Hydrogen occlusion was researched in this mechanical polishing process and hydrogen-free mechanical polishing process was invented.

3. A method for eliminating abrasive grain contaminated layer and damaged layer caused by centrifugal barrel polishing was invented by adopting an electropolishing process (chemical polishing is also applicable) in combination with continuous oxidation process.

4. A simple surface-treating process which achieves high accelerating gradient of 30 MV/m was established by the combination of the above-mentioned inventions with a

conventional electropolishing.

5. As a result, a simplified surface-treating process for superconducting cavity without annealing process was developed.

(Page 3, lines 27-29)

These research results dramatically simplify a surface treating process for superconducting cavity. Therefore, it is apparent that the surface treating process developed by the present study leads to much cost reduction such as reduction of equipment investment and treatment time in the production of 20,000 or more superconducting cavities like TESLA.

Hydrogen Absorption in Electropolishing of Niobium

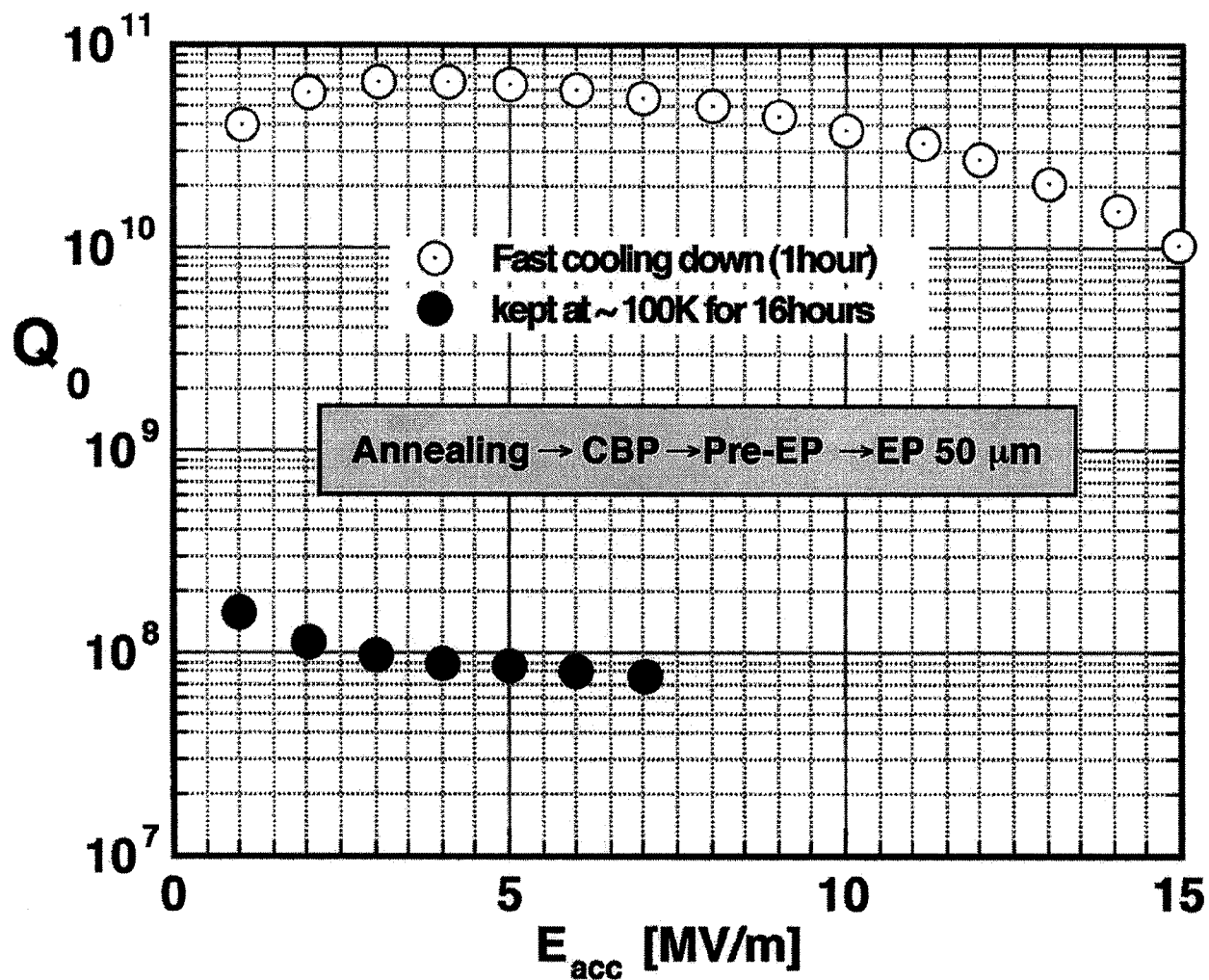
**Accelerator Laboratory,
High Energy Accelerator Research Organization (KEK)**

Tamao HIGUCHI and Kenji SAITO

Contents

- 1. Introduction – hydrogen Q-disease, parametrization**
- 2. What is the real cause for hydrogen Q-disease?**
- 3. Surface defects and absorption of hydrogen**
- 4. Solution for the hydrogen Q-disease**
- 5. Conclusion**

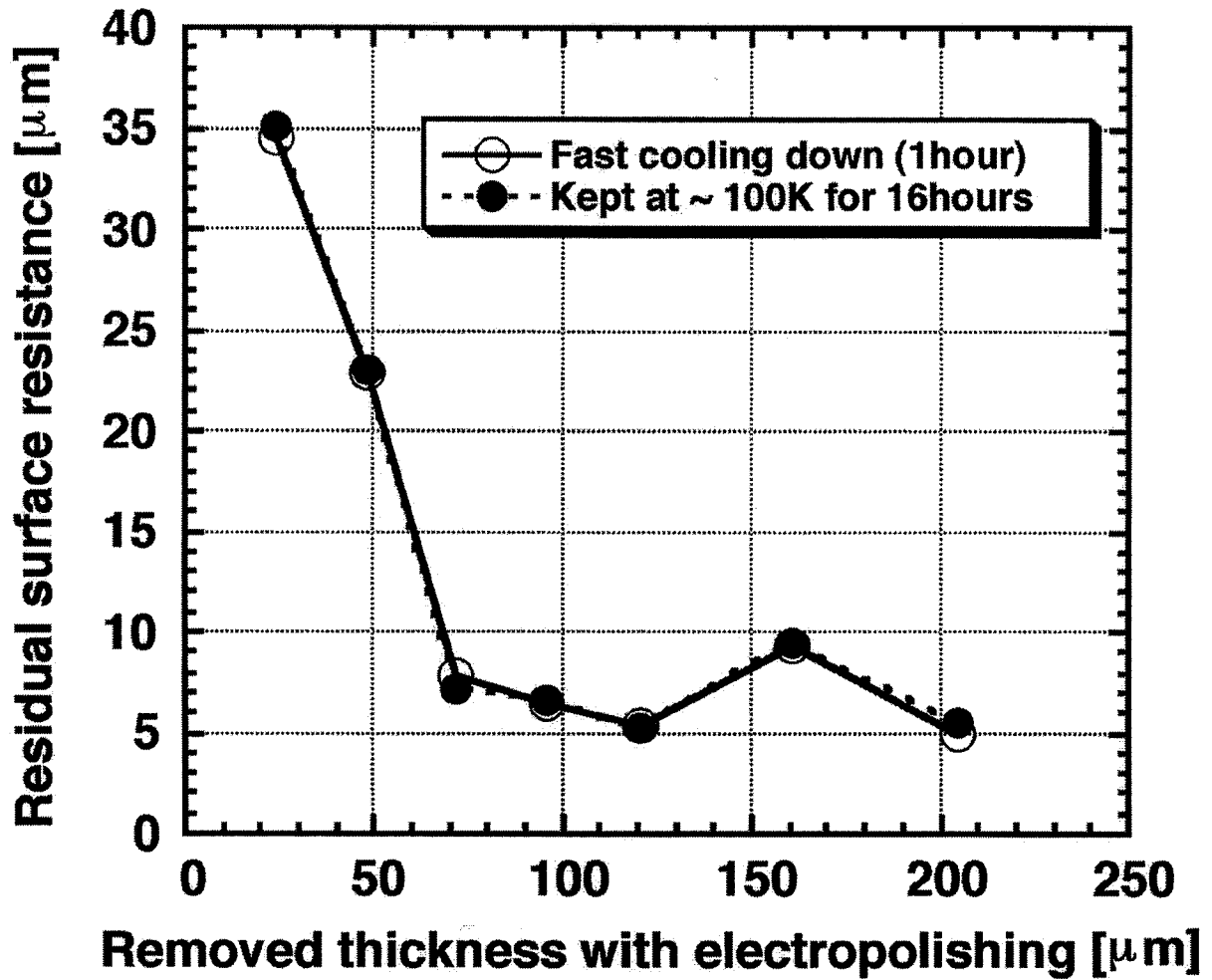
Introduction : Hydrogen Q-disease



Motivation: need better cavity production scheme for future accelerator

Phenomenon: Increased surface resistance

Parametrization: How to evaluate the degree of seriousness

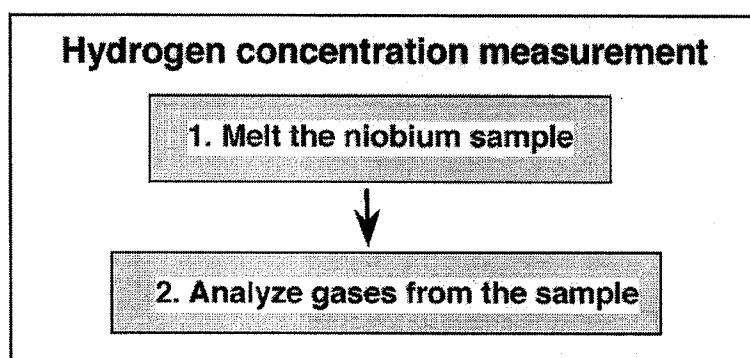


**No hydrogen Q-disease after electropolishing
(even after removing as much as 200 μm)**

What is the real cause for hydrogen Q-disease?

Treatment	H concentration (Nb test sample)	Increase in R_{res} (Nb cavity)
Annealing Nbsheet (750 °C, 3 hours)	less than 1 ppm	(preparation)
Electric discharge machining	5.2 ppm	(preparation)
Annealing samples (750 °C, 3 hours)	0.5 ppm	(preparation)
Dipping in electropolishing solution	6.0 ppm	+ 5.9 n Ω
Electropolishing	0.1 ppm	- 0.7 n Ω

sample size: 2.5 mm x 10 mm x 10 mm

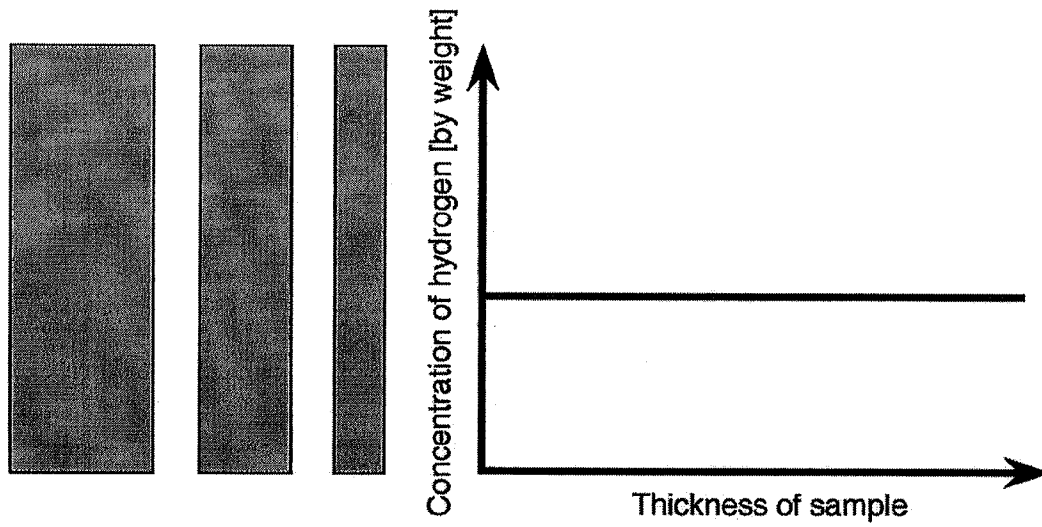


What is responsible for the hydrogen absorption?

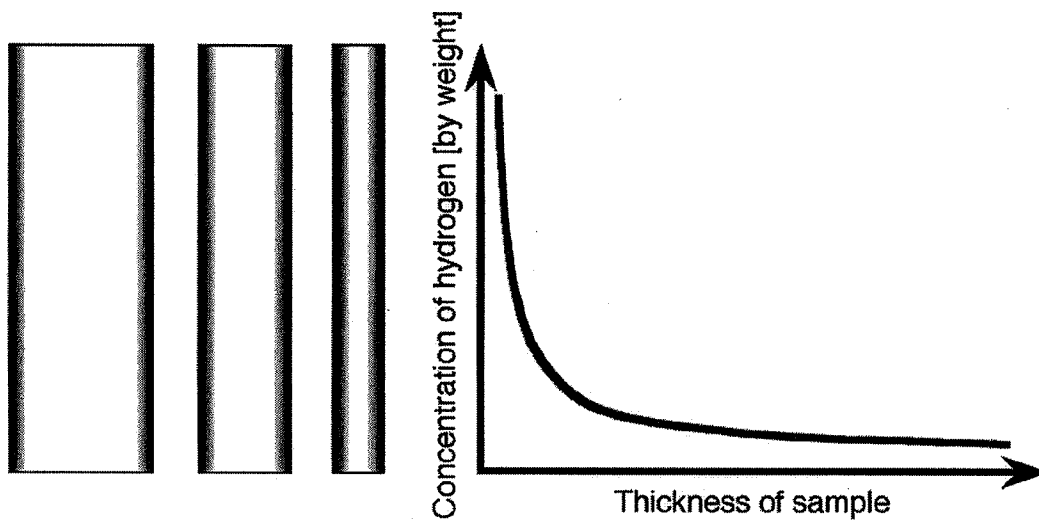


- 1) Electropolishing would not cause hydrogen pick-up**
- 2) Test result on small samples and that on cavities agree**

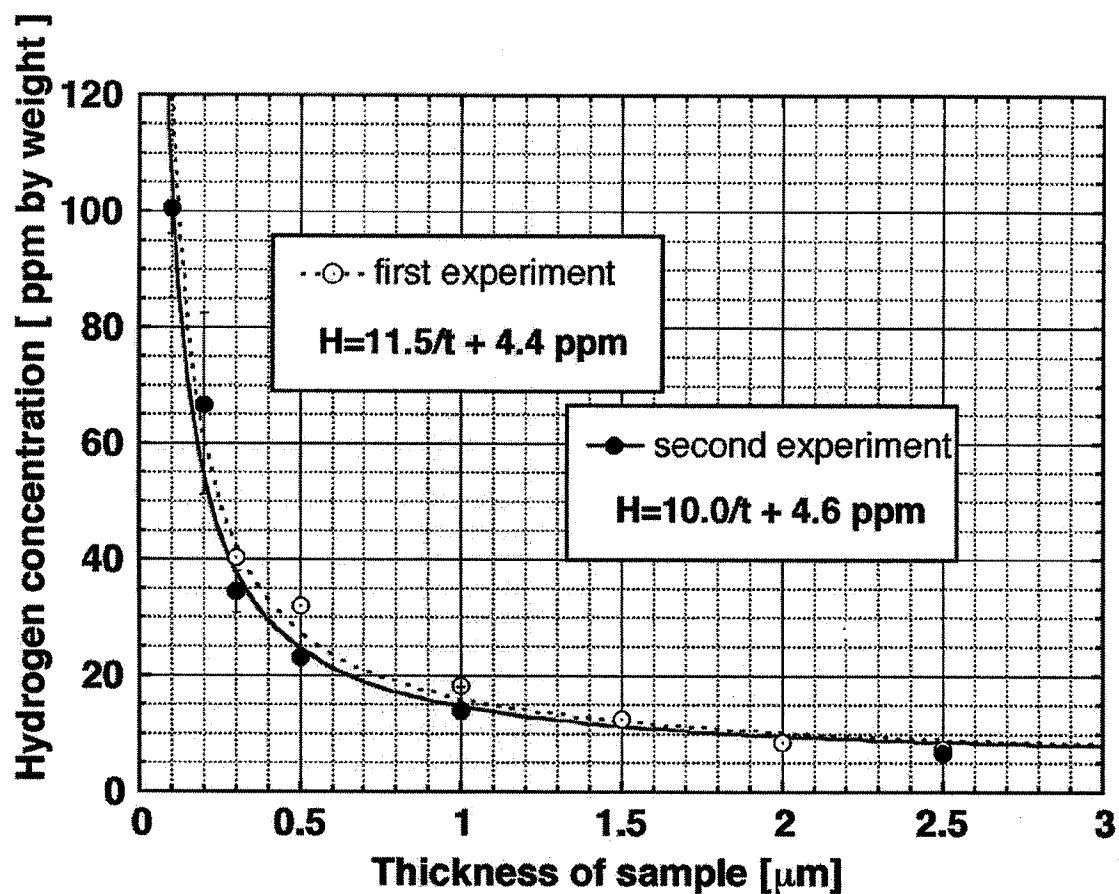
Hydrogen uniformly distributed



Hydrogen concentrated near the surface area

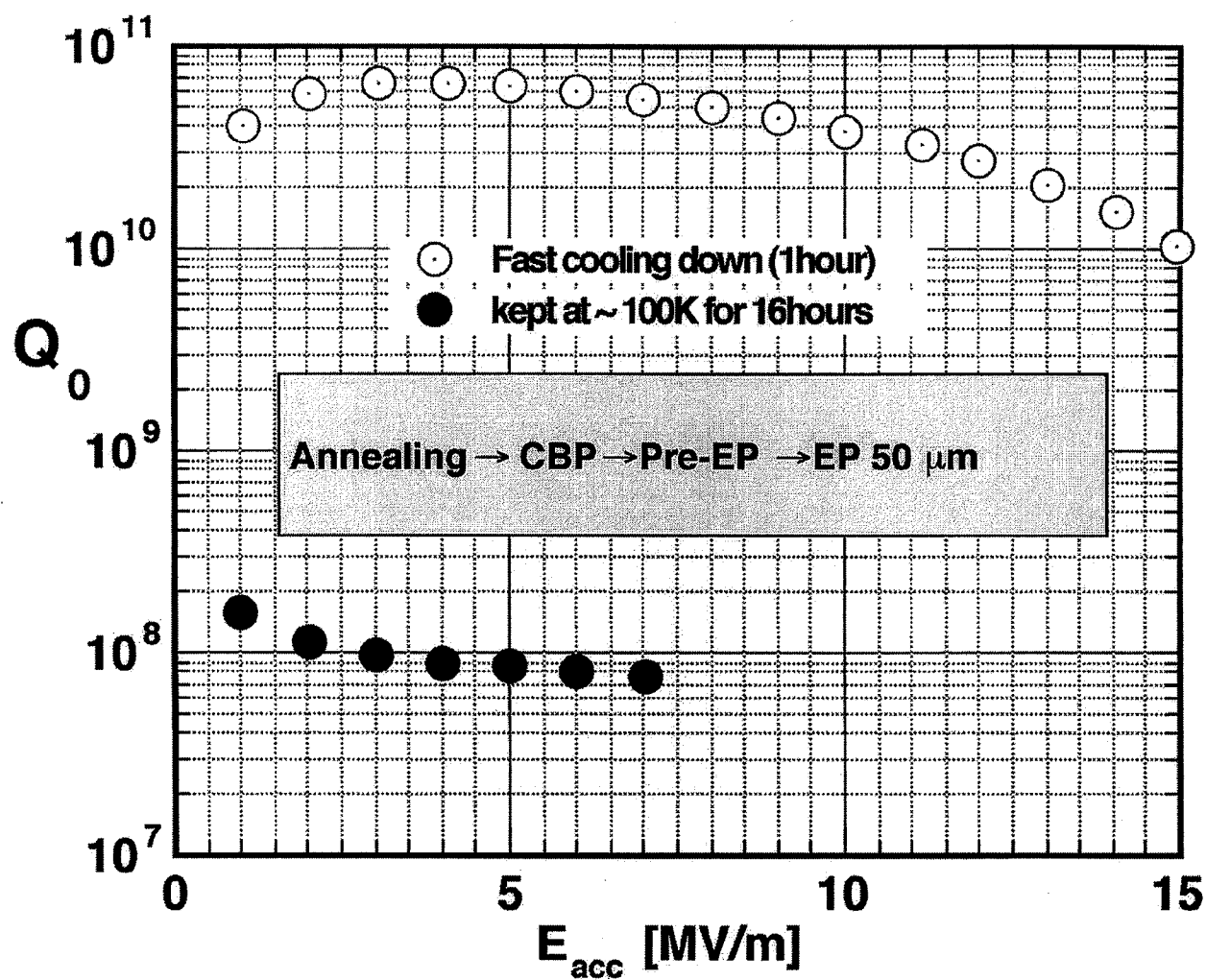


Surface defects and absorption of hydrogen



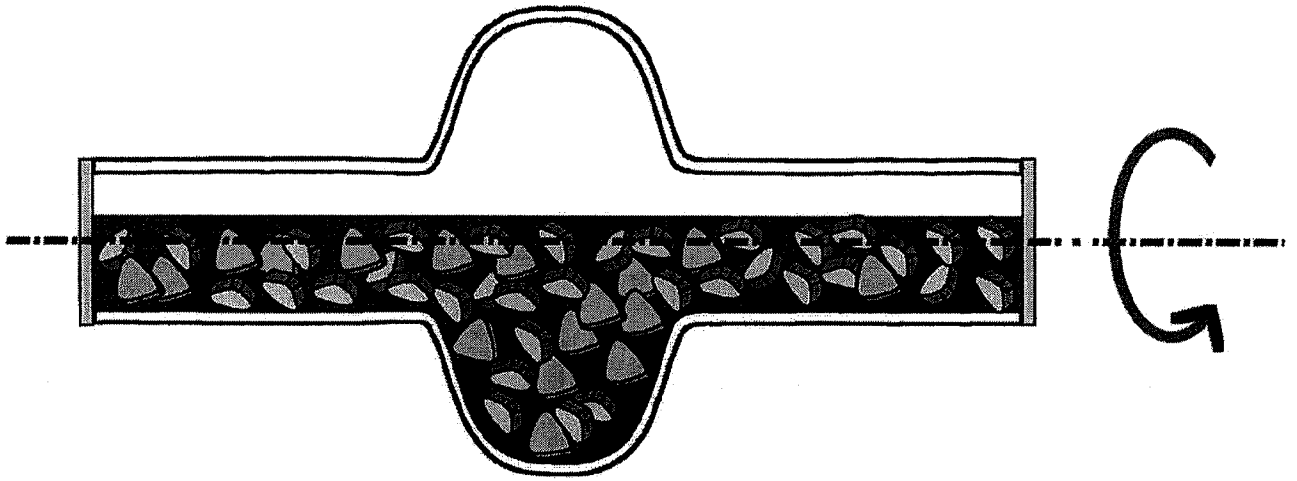
Hydrogen seems to be trapped on the surface defects

What is the real cause for hydrogen Q-disease?

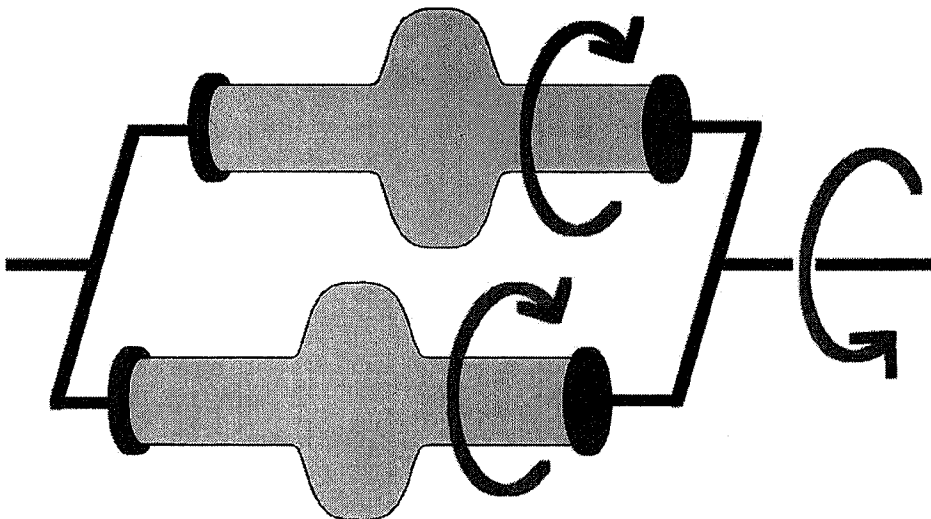


**Hydrogen Q-disease was caused by
a combination of CBP and electropolishing**

Barrel Polishing



Centrifugal Barrel Polishing



Liquid used in CBP process (4 hours)	Hydrogen concentration	Comments
Annealing (750°C, 3 hours)	1.0 ± 0.2 ppm	(Pre treatment)
Demineralized water + detergent	78.0 ± 2.9 ppm	Standard composition
Demineralized water	79.1 ± 5.0 ppm	No detergent
No liquid	(10.9 ± 0.8) ppm	(too slow polishing)
Propanol (C_3H_7OH)	49.4 ± 2.2 ppm	No water
Fluorine-based liquid: FC-77 (mixture of C_8F_{18} and $C_8F_{16}O$)	4.6 ± 2.2 ppm	No hydrogen

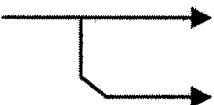
sample size: 1.0 mm x 2.5 mm x 147 mm


To reduce the amount of hydrogen absorption in niobium,




**Use polishing liquid that contains no hydrogen
(FC-77, for example)**

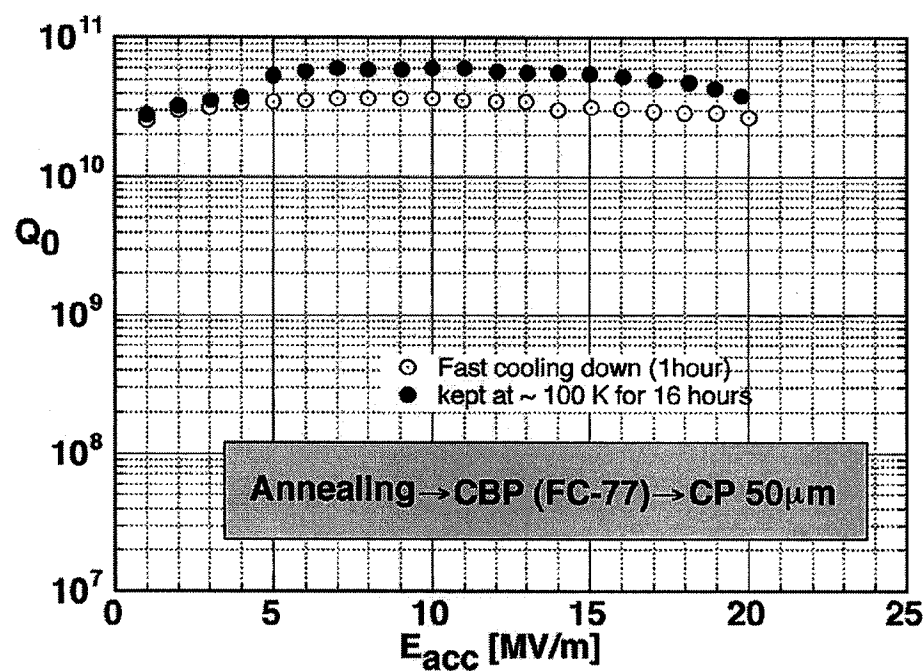
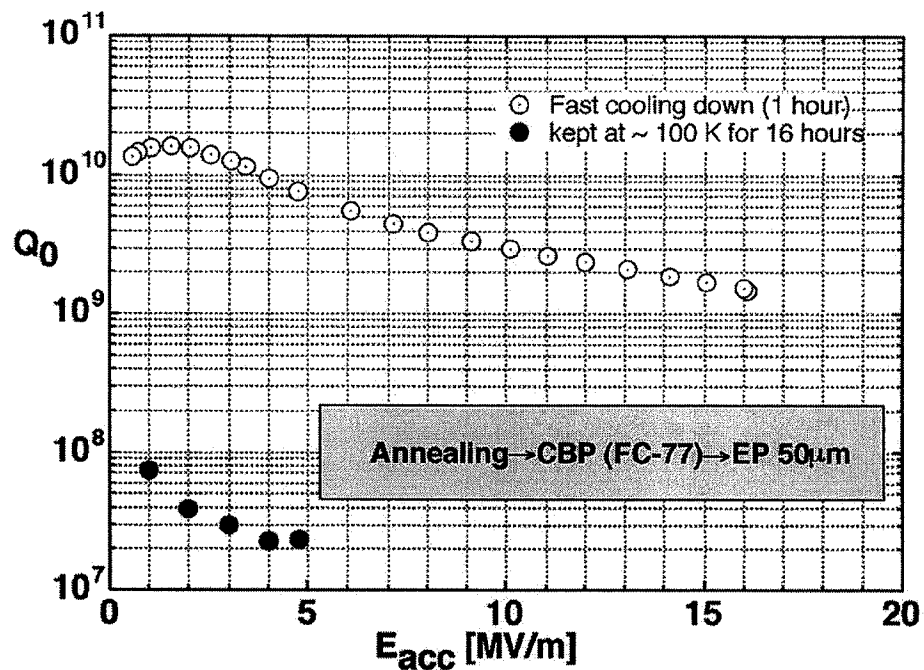
For hydrogen-free preparation

1) CBP (FC-77)  **electropolishing**
chemical polishing

2) CBP (FC-77 + O₃)  **electropolishing**

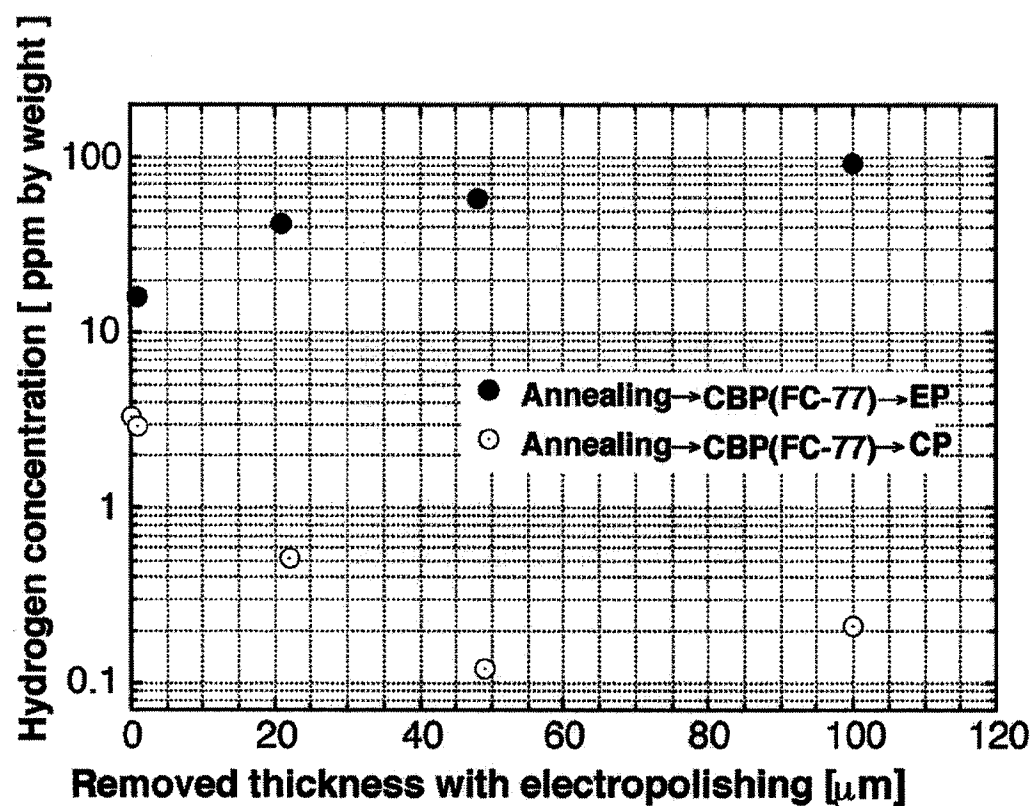
3) CBP (FC-77)  **electropolishing**
(with nitric acid as oxidizer)

4) CBP (FC-77)  **chemical polishing**  **electropolishing**



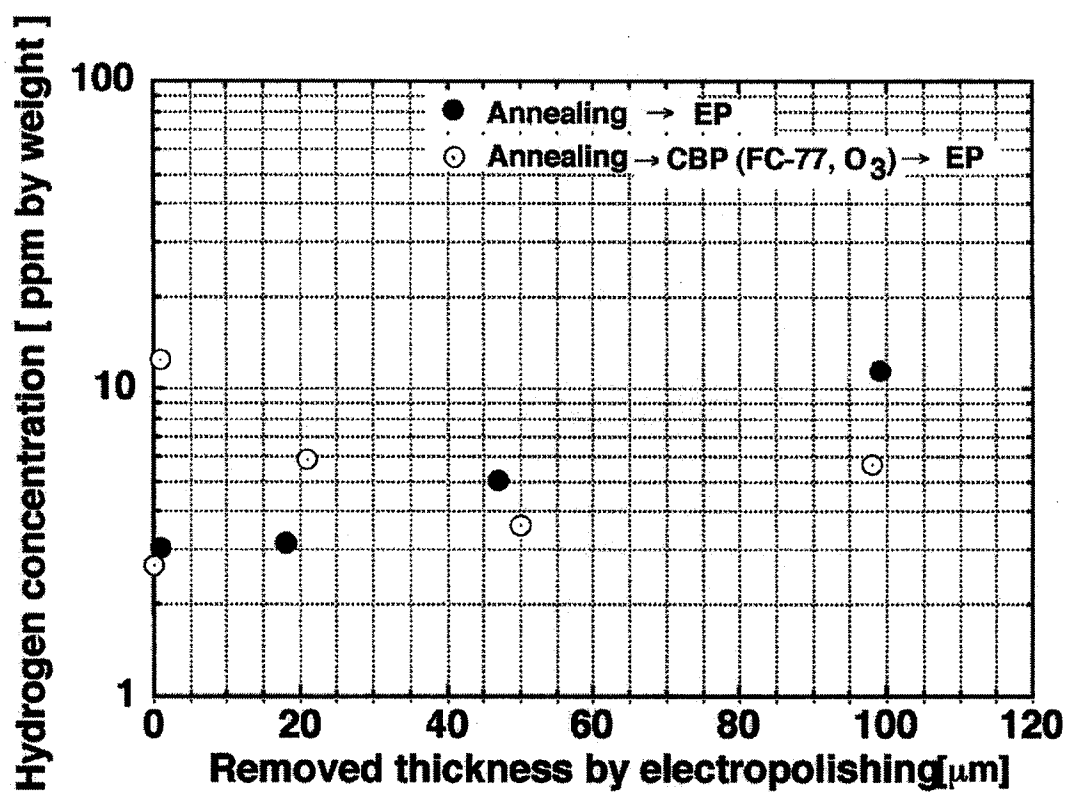
Annealing → CBP (FC-77) → EP:	Hydrogen Q-disease
Annealing → CBP (FC-77) → CP:	No Hydrogen Q-disease

Solution for the Hydrogen Q-disease

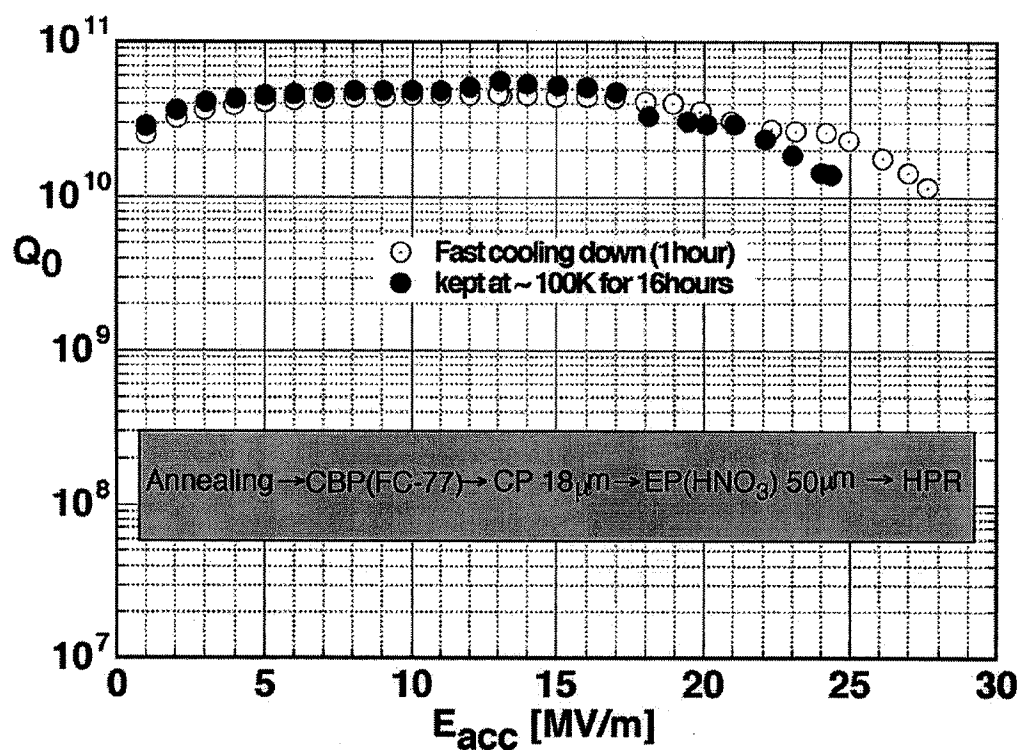


Annealing → CBP(FC-77) → EP : Hydrogen Absorption
Annealing → CBP(FC-77) → CP : No Hydrogen Absorption

Solution to the Hydrogen Q-disease problem

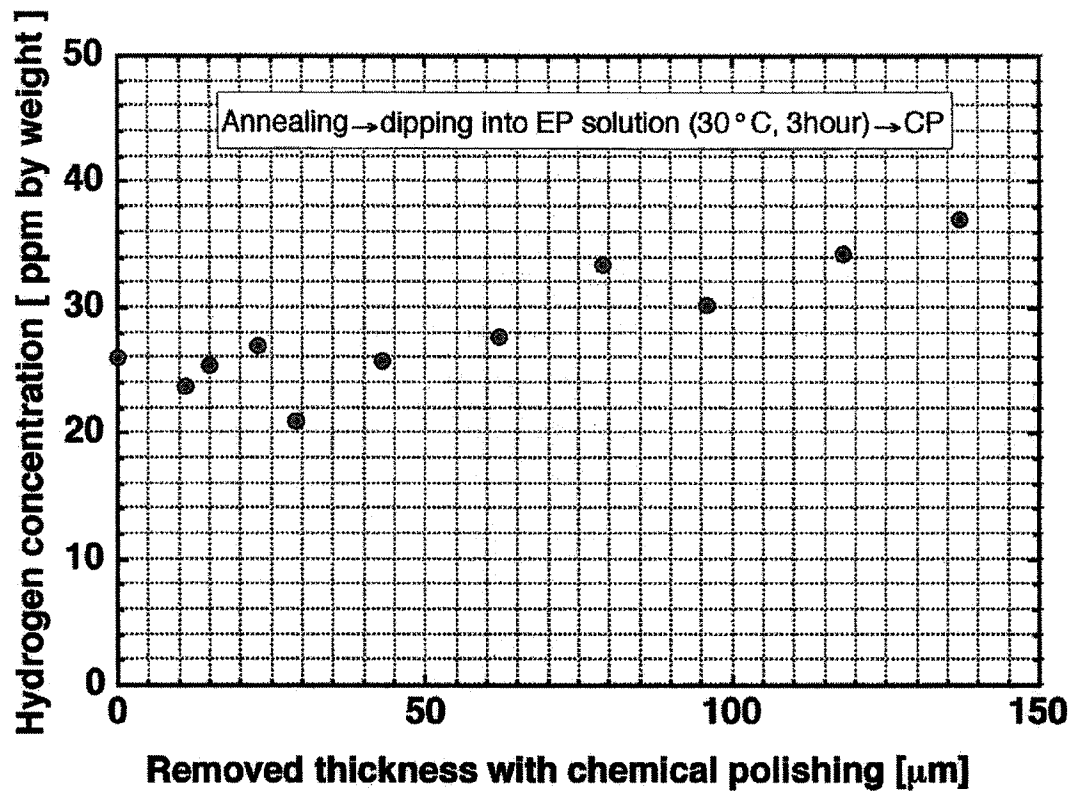


Oxidation effect stays on after electropolishing



Annealing → CBP(FC-77) → slight CP → EP(HNO₃): No Hydrogen Q-disease

Once hydrogen absorption occurred in earlier stage, it will continue during chemical polishing process.



Conclusions

- 1) Absorbed hydrogen becomes a seed for successive hydrogen absorption in chemical process that follows.
- 2) Polishing process with continuous oxidation is a way for hydrogen-free preparations.